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Preparation and characterization of diatomite/silica composite humidity control material by partial alkali dissolution

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ABSTRACT

The diatomite/silica composite humidity control material (DE/S) was prepared via partial alkali dissolution using diatomite (DE) as silicon source. The samples obtained were characterized for reaction mechanism and pore structure. The humidity control performances and reusability experimental of samples were analyzed. The results show that DE/S has better humidity control performance than DE under different relative humidity and temperature. DE/S shows good reusability. Humidity control performance of DE is significantly improved by composite material generated during the alkali dissolution as increasing of microporous and mesoporous, which form more reasonable pore size distribution and increase the structure heterogeneity.

1. Introduction

Interior air environment with comfortable humidity is required nowadays on many occasions as the development of technology and society [1,2]. Both damp and dry environment have adverse influence on human health and livelihood. Generally, air-conditioning equipment has been used to control the relative humidity, which is not economic [3]. Humidity control material is superior to conventional methods as non-energy consumption.

Diatomite (DE) is made up of fossilized diatom derived from the deposition of single-cell water plant [4,5]. DE has many useful features such as high porosity and bulk volume, chemical resistance, large specific surface area and adsorption capacity [4]. DE has been investigated as humidity control material [6]. Specifically, it has been widely used as interior wall material as the development of diatom ooze in recent years.

Silica is a significant raw material in many industries such as coating, paint, rubber and plastic for its predominant physico-chemical property. Conventional fumed silica has the disadvantage of expensive manufacture cost. Precipitate silica which uses non-metallic minerals as silicon source has attracted wide attention for its low price and simple technique. Alkali dissolution is most common method to prepare sodium silicate, which is precursor of precipitate silica.

However, until now, few attentions have focused on the preparation of diatomite/silica composite humidity control material (DE/S). The ensuing paper is an attempt to investigate the preparation and characterization of DE/S. In this study, the composite material was prepared via partial alkali dissolution. XRD, FTIR, SEM, ICP-OES, low temperature nitrogen adsorption, mercury intrusion porosimetry (MIP) and fractal dimensions were employed to characterize the reaction mechanism and pore structure of as-prepared sample, and the humidity control performance was investigated.

2. Material and methods

The DE used in this investigation was collected from Changbai deposit in Jilin Province of China. The main chemical compositions of DE were listed as follow: SiO_2 86.97%, Al_2O_3 3.76%, Fe_2O_3 2.13%. Sodium hydroxide and sulfuric acid were purchased from Beijing Chemical Reagent Company (Beijing, China).

The DE/S was prepared as follow: 20.00 g DE and 7.73 g sodium hydroxide were dispersed in the beaker with 100 ml distilled water at the water bath of 80 °C for 30 min. Then sulfuric acid (50 wt.%) was added to the beaker with a ratio of 5r/min using a peristaltic pump. After stewing for further 20 min, the product was collected and centrifuged. Then the sediment was collected and dried in an oven at 105 °C for 12 h.

The XRD patterns of the samples were performed via X-ray diffractometer (Bruker D8 Advance, Germany) employing CuKα radiation at a scan speed of 4°/min in the 2θ range of 5–80°.

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ICP-OES (Agilent 7500ce, USA) was used to analyze the element concentrations of the filtrate. FTIR (Bruker Vertex70, Germany) and SEM (Hitachi S-4800, Japan) were used to further confirm the generation of composite. The pore structure analysis based on nitrogen gas adsorption/desorption was carried out by a specific surface area analyzer (Quantachrome Autosorb-iQ, USA). The surface area was measured using BET method and t-plot method. The pore volume and pore distribution were tested by BJH method using adsorption curve. MIP was carried out with a mercury porosimeter (Micromeritics AutoPore IV9500, USA). The fractal dimensions were calculated by FHH model using adsorption curves.

Humidity control performances were determined by the method of ISO12571-2006 with high-low temperature testing chest (GDW-300, China). To examine the moisture adsorption/desorption abilities, weight change of DE and DE/S were monitored. The moisture content of samples was calculated by the following equation:

$$M = \frac{m_t - m_0}{m_0} \times 100\%$$

where M is moisture content, m_0 is the initial weight of the dried sample, and m_t is the weight of the sample at time t.

3. Results and discussion

Fig. 1(a) shows the XRD patterns of DE and DE/S. Seen from the XRD results, an essentially amorphous phase which is the main phase composition of diatom and silica in the 2θ range of 20–25° are observed in the DE and DE/S. A quartz phase (PDF 46-1045) and muscovite phase (PDF 07-0025) as impurity minerals are found in XRD patterns of these samples. As Fig. 1(b) shown, the broad intense band centered at 3433 cm^{-1} and narrow band at 1638 cm^{-1} can be related with O—H stretching vibration and O—H bending vibration, respectively. The bands at 1095, 798 and 465 cm^{-1} originate from the vibrations of the Si—O—Si bonds. Compared Fig. 1(c) with Fig. 1(d), flocculent spherical silica particles coat on the DE surface, and surface roughness increases. To further analyze the reaction mechanism, the filtrate (3.26 L) was collected, and the chemical compositions were analyzed by ICP-OES. The main element concentrations in the filtrate are sodium (1407.49 mg/L) and sulfur (999.33 mg/L), which basically agree with the initial dosage of sodium hydroxide and sulfuric acid. Part of diatom reacts as silicon source, and the rest diatom works as skeleton of composite material. The main chemical reactions of preparation silica are as follow:

$$2NaOH + SiO_2 \rightarrow Na_2SiO_3 + H_2O \quad (1)$$

$$Na_2SiO_3 + H_2SO_4 \rightarrow H_2SiO_3 + Na_2SO_4 \quad (2)$$

$$H_2SiO_3 \rightarrow SiO_2 + H_2O \quad (3)$$

As Fig. 2(a) shown, the nitrogen gas adsorption/desorption isotherms of DE and DE/S are in accordance with the typical IV curves with H3 hysteresis loops according to the classification of IUPAC [7]. And clear capillary condensation occurs approximately at the relative pressure p/p_0 of 0.6–0.9. The hysteresis loops for this type of isotherm confirm the existence of mesoporous in these samples.

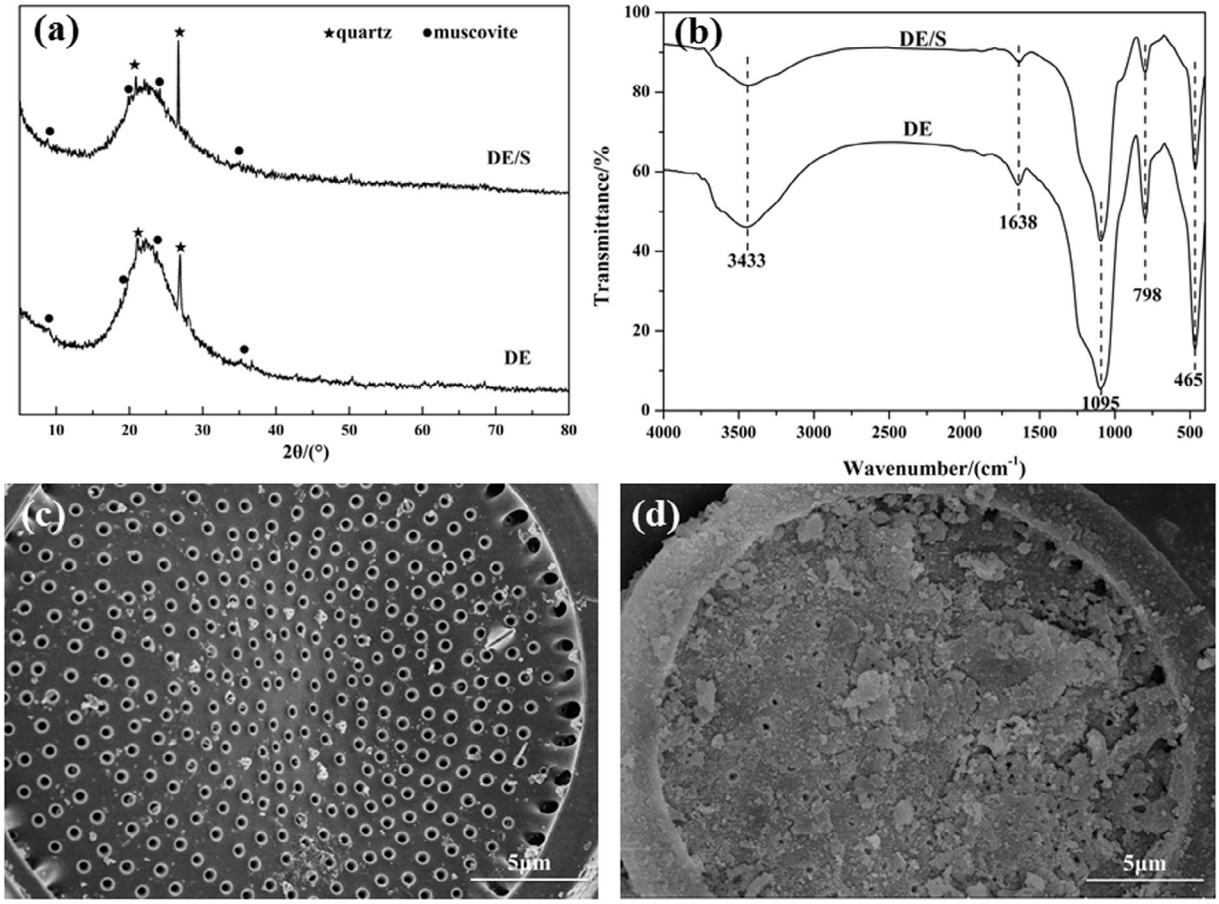

Fig. 1. XRD patterns of DE and DE/S (a), FTIR spectra of DE and DE/S (b), SEM image (×5000) of DE (c), SEM image (×5000) of DE/S (d).